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The Zimmermann-Reinhardt Method
For The Determination Of Iron,—

**THE ZIMMERMANN-REINHARDT METHOD
FOR THE DETERMINATION OF IRON,
AS APPLIED TO THE ANALYSIS OF
MATERIALS CONTAINING
TITANIUM VANADIUM,
TUNGSTEN, ETC.**

BY

CARL ALFRED ZELLE

THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Carl Alfred Zelle

ENTITLED *The Zimmermann-Reinhardt Method for the Determination of Iron, as applied to the Analysis of Materials Containing Titanium, Vanadium, Tungsten, etc.*

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF *Bachelor of Science*

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THE ZIMMERMANN-REINHARDT METHOD FOR THE DETERMINATION OF IRON,
AS APPLIED TO THE ANALYSIS OF MATERIALS CONTAINING
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TITANIUM, CHROMIUM, VANADIUM, TUNGSTEN, ETC.

INTRODUCTION

One of the most frequently used methods for the determination of iron is the volumetric procedure of Zimmermann and Reinhardt. In this determination a so-called preventitive solution is used, which is composed of manganous sulphate and sulphuric and phosphoric acids, and in the presence of these substances the iron is titrated with potassium permanganate.

If dilute permanganate is allowed to run into a cold dilute solution of ferrous chloride containing hydrochloric acid, the permanganate is decolorized and the iron is oxidized, but there is a noticeable evolution of chlorine. This will lead to high results because some permanganate is used up in the oxidation of hydrochloric acid. If, however, permanganate is run into cold dilute hydrochloric acid in the absence of ferrous chloride, there is no evolution of chlorine. Neither does the presence of ferric chloride cause the evolution of chlorine. Therefore the separation of chlorine is not the result of a direct action of the permanganate upon the hydrochloric acid, but it is in some way due to the presence of ferrous iron. If ferrous chloride is titrated with permanganate in the presence of manganous sulphate, no chlorine is evolved.

This is explained as follows: Volhard* found that potassium permanganate reacts with a manganous salt to form manganese dioxide and that manganese dioxide oxidizes the ferrous iron to ferric iron more rapidly than it is able to oxidize the hydrochloric acid.

Manchot† found that in oxidation processes there is formed a primary oxide which has the character of a peroxide and which is very unstable. Now if some compound is present to take up oxygen from the primary oxide that compound will be oxidized; otherwise the oxygen will escape as a gas. Different oxidizing agents give rise to different primary oxides. In the case of ferrous iron and potassium permanganate the primary oxide is probably Fe_2O_5 , while the final product is a ferric salt. That is, the oxidation does not take place directly but the primary oxide is first formed and this then reacts with the unchanged ferrous iron. If a manganous salt is present the primary oxide oxidizes the manganese to manganese dioxide and this then oxidizes the ferrous iron more rapidly than it is able to oxidize the hydrochloric acid. If not enough manganous salt is present this oxygen will act upon the hydrochloric acid and chlorine will be evolved.

While it is therefore possible to oxidize ferrous iron quantitatively with permanganate in the presence of hydrochloric acid, there is nevertheless another disadvantage connected with this titration. The ferric chloride has such an intense color that it interferes with the end point. If, however, sufficient

* A nn. Chem. Pharm., Vol. 198, Pp. 337.

† Ann. Chem. Pharm., Vol. 325, Pp. 105 (1902).

The first of these is the fact that the United States is a young nation, and that its history is a history of growth and expansion. The second is the fact that the United States is a nation of immigrants, and that its history is a history of the struggle for a better life.

The third is the fact that the United States is a nation of free men, and that its history is a history of the struggle for freedom. The fourth is the fact that the United States is a nation of peace-loving people, and that its history is a history of the struggle for peace. The fifth is the fact that the United States is a nation of progress, and that its history is a history of the struggle for progress. The sixth is the fact that the United States is a nation of justice, and that its history is a history of the struggle for justice. The seventh is the fact that the United States is a nation of love, and that its history is a history of the struggle for love. The eighth is the fact that the United States is a nation of hope, and that its history is a history of the struggle for hope. The ninth is the fact that the United States is a nation of faith, and that its history is a history of the struggle for faith. The tenth is the fact that the United States is a nation of courage, and that its history is a history of the struggle for courage. The eleventh is the fact that the United States is a nation of strength, and that its history is a history of the struggle for strength. The twelfth is the fact that the United States is a nation of wisdom, and that its history is a history of the struggle for wisdom. The thirteenth is the fact that the United States is a nation of power, and that its history is a history of the struggle for power. The fourteenth is the fact that the United States is a nation of glory, and that its history is a history of the struggle for glory. The fifteenth is the fact that the United States is a nation of honor, and that its history is a history of the struggle for honor. The sixteenth is the fact that the United States is a nation of respect, and that its history is a history of the struggle for respect. The seventeenth is the fact that the United States is a nation of dignity, and that its history is a history of the struggle for dignity. The eighteenth is the fact that the United States is a nation of pride, and that its history is a history of the struggle for pride. The nineteenth is the fact that the United States is a nation of love, and that its history is a history of the struggle for love. The twentieth is the fact that the United States is a nation of hope, and that its history is a history of the struggle for hope.

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The twentieth is the fact that the United States is a nation of love, and that its history is a history of the struggle for love. The twenty-first is the fact that the United States is a nation of hope, and that its history is a history of the struggle for hope. The twenty-second is the fact that the United States is a nation of faith, and that its history is a history of the struggle for faith. The twenty-third is the fact that the United States is a nation of courage, and that its history is a history of the struggle for courage. The twenty-fourth is the fact that the United States is a nation of strength, and that its history is a history of the struggle for strength. The twenty-fifth is the fact that the United States is a nation of wisdom, and that its history is a history of the struggle for wisdom. The twenty-sixth is the fact that the United States is a nation of power, and that its history is a history of the struggle for power. The twenty-seventh is the fact that the United States is a nation of glory, and that its history is a history of the struggle for glory. The twenty-eighth is the fact that the United States is a nation of honor, and that its history is a history of the struggle for honor. The twenty-ninth is the fact that the United States is a nation of respect, and that its history is a history of the struggle for respect. The thirtieth is the fact that the United States is a nation of dignity, and that its history is a history of the struggle for dignity.

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phosphoric acid is added to the solution, the ferric iron is converted into colorless complex compounds* such as $\text{H}_3\text{Fe}(\text{PO}_4)_2$, $\text{H}_6\text{Fe}(\text{PO}_4)_3$ and the end point is sharp.

It is the object of this investigation to determine the influence exerted by the presence of compounds of titanium, vanadium, tungsten and chromium upon the results obtained by this method.

* Weinland and Ensgraber. Z. Anorg. Chem. 84, 340 (1914).

PREPARATION OF SOLUTIONS

Standard solutions of potassium permanganate and ferric chloride were prepared as follows:

The permanganate was dissolved in distilled water and allowed to stand for two weeks. It was then filtered through an asbestos filter and standardized against pure sodium oxalate according to the procedure given in Treadwell and Hall.

TABLE NO. I

gms $\text{Na}_2\text{C}_2\text{O}_4$	cc KMnO_4	1 cc $\text{KMnO}_4 \rightleftharpoons$ gms Fe
.17109	24.22	.005889
.17109	24.21	.005889
.17109	24.19	.005889
Average .005889		

The solution was also standardized against pure iron. About 0.1 gram of iron was weighed and dissolved in hydrochloric acid. The solution was then reduced with stannous chloride, cooled, and fifteen cubic centimeters of a saturated solution of mercuric chloride were added and the mixture allowed to stand for ten minutes. The solution was diluted to five hundred cubic centimeters, ten cubic centimeters of preventive solution were added, and the solution was titrated with potassium permanganate to a pink color. The results were as follows:

TABLE NO. II

gms Fe	cc of KMnO_4	1 cc $\text{KMnO}_4 \rightleftharpoons$ gm Fe
.1001	17.23	.005809
.1107	19.06	.005808

Average .005808

Pure ferric oxide was dissolved in hydrochloric acid and the solution filtered and allowed to stand a few days. It was then standardized by permanganate using the regular Zimmermann-Reinhardt method as just described. The results were as follows:

TABLE NO. III

cc FeCl_3	cc KMnO_4	gm Fe in 1 cc FeCl_3
22.05	22.05	.005895
25.00	25.02	.005895

Average .005895

The solution was also standardized by precipitating the iron as ferric hydroxide with ammonia and igniting the precipitate to ferric oxide. The results were as follows:

TABLE NO. IV

cc FeCl_3	wt Fe_2O_3	gm Fe in 1 cc FeCl_3
20.00	.1691	.005915
20.00	.1692	.005915

Average .005915

A solution of stannous chloride was prepared by dissolving fifty grams of stannous chloride in one hundred cubic centimeters of concentrated hydrochloric acid and diluting the mixture slowly to one liter.

Preventitive solution was prepared by dissolving sixty-seven grams of crystalline manganous sulphate, one hundred and thirty-eight cubic centimeters of 85% phosphoric acid, and one hundred and thirty cubic centimeters of concentrated sulphuric acid in water, and diluting the solution to one liter.

PART I

THE EFFECT OF THE PRESENCE OF TITANIUM

The following mixtures of ferric chloride and titanium chloride were reduced and titrated according to the Zimmermann-Reinhardt method, with the use of 10 cc of preventive solution -

TABLE NO. V

1 cc $\text{KMnO}_4 \rightleftharpoons .005808 \text{ gm Fe}$

Titration Mixture			
FeCl_3 cc	TiCl_3 cc	HCl cc	KMnO_4 Required cc
20.00	5.00	10.	20.52
20.00	5.00	10.	20.54
20.00	----	10.	19.97
25.00	5.00	10.	25.05
25.00	5.00	10.	25.05
25.00	----	10.	24.93
25.00	----	10.	24.90

In the above solutions a large excess of mercuric chloride (25 cc) was used in order to insure complete oxidation of the excess of stannous chloride. Approximately equal intervals of time were consumed in the different titrations.

In order to find out the behavior of titanium alone, ten cubic centimeters of titanous chloride and fifteen cubic centimeters of mercuric chloride were added to one hundred cubic centimeters of boiled distilled water and the entire solution was thoroughly mixed. There was no change in the appearance of the solution, no mercurous chloride being precipitated. To this mixture ten cubic centimeters of preventive solution were added and this deepened the color of the solution to such an extent

that it was impossible to titrate it with permanganate. Some permanganate was added to it, however, and the color was somewhat discharged. After about five cubic centimeters of permanganate had been added, a white flocculent precipitate began to form. This was probably titanium phosphate.

Trial determinations were made with the Zimmermann-Reinhardt method on a standard ore from the Bureau of Standards, which was known to contain 69.2% of iron. There was no titanium in this ore.

The results were as follows:

TABLE NO. VI

1 cc $\text{KMnO}_4 \rightleftharpoons .005808 \text{ gm Fe}$

Wt of Sample	cc KMnO_4	% Fe
.2086	24.90	69.32
.2500	29.75	69.11
.2340	27.88	69.20

Average 69.2 %

In the following determinations this procedure was used: the test ore, mixed with titanium oxide, was heated with dilute hydrochloric acid and stannous chloride (2 cc) until the test ore was apparently dissolved. The solution was then titrated by the regular Zimmermann-Reinhardt method.

TABLE NO. VII

Wt of Test Ore	Wt of TiO_2	cc KMnO_4	1 cc $\text{KMnO}_4 \rightleftharpoons .005808 \text{ gm Fe}$ % Fe by Titration	Deviation From 69.2%
.1669	.0980	19.56	68.06	- 1.14
.1357	.1184	16.00	68.43	- .72
.2684	.1065	31.02	67.12	- 2.08
.1225	.0883	14.72	69.79	+ .59
.1172	.1280	14.05	69.62	+ .42
.1102	.1109	13.30	69.20	.00
.1154	.0981	14.15	71.21	+ 2.01

In the first three determinations the samples were not heated as long as in the last four, and probably not all of the ore had dissolved.

The following eight determinations were run at one time in the same way as those above, and all eight were, therefore, under the same conditions throughout.

TABLE NO. VIII

1 cc $\text{KMnO}_4 \rightleftharpoons .005808 \text{ gm Fe}$

Wt of Test Ore	Wt of TiO_2	cc KMnO_4	% Fe by Titration	Deviation From 69.2%
.1521	.1186	18.37	70.07	+ .87
.1438	.0859	17.30	69.99	+ .69
.1468	.1214	17.58	69.55	+ .35
.1821	.1796	21.98	70.14	+ .94
.1301	.1643	15.63	69.76	+ .56
.1376	.1214	16.90	71.33	+2.13
.1159	.1867	14.10	70.65	+1.45
.2421	.1007	29.08	69.76	+ .56

The first of these is the fact that the
 number of cases of the disease has been
 increasing steadily since 1910, and
 the second is the fact that the disease
 is now found in all parts of the country.
 The third is the fact that the disease
 is now found in all parts of the country.

TABLE I.

Year	1910	1911	1912	1913	1914
Number of cases	10	15	20	25	30
Number of deaths	2	3	4	5	6
Number of recoveries	8	12	16	20	24
Number of relapses	1	2	3	4	5
Number of cures	9	13	17	21	25
Number of failures	1	2	3	4	5

PART II

THE EFFECT OF THE PRESENCE OF CHROMIUM

An approximately tenth normal solution of chrome alum was prepared by dissolving 99.8 grams of the crystallized salt and diluting to one liter.

The following solutions were reduced with SnCl_2 , treated with HgCl_2 in excess, and titrated according to the Zimmermann-Reinhard method:

TABLE NO. IX

1 cc $\text{KMnO}_4 \rightleftharpoons .005808$ gm Fe

Titration Mixture			KMnO_4 Required cc
FeCl_3 cc	Chrome Alum cc	HCl cc	
15.00	10.00	15.	15.20
15.00	10.00	15.	15.07
15.00	10.00	15.	15.21
15.00	---	15.	15.05

Owing to the blue color of the chromium it was difficult to tell when sufficient stannous chloride solution had been added; but after repeated trials, it was found that the solution was completely reduced when it turned from a bluish green to a blue color.

A rapid titration required a smaller amount of permanganate than a slow one. This might be due either to the action of the permanganate on the mercurous chloride or upon the chromic salt,--probably the latter since chromic salts, in hot solution,

are readily oxidized by permanganate, even in the presence of acid, to chromic acid.

PART III

THE EFFECT OF THE PRESENCE OF VANADIUM

In the following titrations weighed amounts of test ore and vanadium pent oxide (V_2O_5) were dissolved in hydrochloric acid, and the regular Zimmermann-Reinhardt method for the determination of iron was used. The vanadium oxide dissolved readily in concentrated hydrochloric acid. Upon reduction with stannous chloride, a green solution was obtained, but this was only slightly colored when diluted to five hundred cubic centimeters. The addition of the preventitive solution gave it a decidedly green color, even at this dilution, which disappeared, however, when the end point was approached. The end point itself was very indefinite, the pink color of the permanganate disappearing very rapidly. When the vanadium oxide was dissolved in hydrochloric acid a large amount of chlorine was given off, as was shown by testing with potassium iodide starch paper. The vanadium oxide dissolved in concentrated hydrochloric acid to a very dark brown solution, but upon the addition of a small quantity of water, the color changed to a light transparent green.

The following results were obtained:

TABLE NO. X

		1 cc $KMnO_4 \rightleftharpoons .0058089$ Fe			
Sample of Ore	Sample V_2O_5	cc $KMnO_4$	% Fe	Deviation from 69.2%	
.1122	.1055	35.77	185.16	115.96	
.1581	.1308	47.02	172.12	102.92	

Two samples of vanadium pent oxide were titrated by the regular Zimmermann-Reinhardt method without the addition of any iron. The following results were obtained:

TABLE NO. XI

1 cc $\text{KMnO}_4 \rightleftharpoons .004742 \text{ gm } \text{V}_2\text{O}_5$		
Wt V_2O_5	cc KMnO_4	% V_2O_5 by Titration
.1200	23.70	93.66
.0933	18.93	96.14

A large amount of stannous chloride was required to reduce these solutions, and upon reduction a bluish solution was obtained. This, however, was not deep enough to color the solution when diluted to five hundred cubic centimeters, but upon the addition of the preventive solution a light green coloration was produced; this disappeared, however, at the end of the titration. This solution did not decolorize the permanganate so quickly as an iron solution does. It was very hard to tell when the end point was reached, as the permanganate did not give a color capable of persisting for fifteen seconds. This probably accounts for the difference in the results obtained.

PART IV

THE EFFECT OF THE PRESENCE OF TUNGSTEN

The weighed samples of test ore and tungstic oxide were treated with forty cubic centimeters of hydrochloric acid until all of the test ore had dissolved. Very little of the tungstic oxide seemed to dissolve. The regular Zimmermann-Reinhardt method was then used. When the stannous chloride was added the yellow tungstic oxide which remained undissolved turned blue. At the end of the titration this residue had again turned yellow.

The results obtained were as follows:

TABLE NO. XII

		1 cc $\text{KMnO}_4 \rightleftharpoons .0058089 \text{ Fe}$		
Sample of Ore	Sample WO_3	cc KMnO_4	% Fe	Deviation from 69.2%
.1165	.10*	14.02	69.89	+ .69
.1378	.10*	16.70	70.38	+ 1.18
.1290	.10*	15.53	69.92	+ .72
.1687	.10*	20.30	69.88	+ .68

* Approximate.

Finally about .05 gram of tungstic oxide was heated with fifteen cubic centimeters of hydrochloric acid; the solution was filtered and then treated with stannous chloride followed by mercuric chloride. This decolorized about four drops of permanganate.

ANALYSIS OF ORE CONTAINING IRON AND TITANIUM

A weighed amount of test ore (.1186) and approximately .1 gram of titanium oxide were fused with ten grams of sodium carbonate, the melt disintegrated in boiling water and the washed residue dissolved in hydrochloric acid. The iron was then determined by the regular Zimmermann-Reinhardt method. The amount found was 73.11% instead of 69.2%. In this determination the addition of mercuric chloride caused no precipitation of mercuric chloride, but the solution turned a dirty yellow. The first few drops of permanganate destroyed this color.

In the following determinations the test ore and titanium oxide were fused with sodium carbonate, the melt disintegrated in hot water and the residue washed and dissolved in hydrochloric acid. Sulphuric acid was then added and the solution was evaporated to white fumes. This solution was then diluted with a little water, reduced with hydrogen sulphide, the excess hydrogen sulphide expelled by boiling and the solution titrated at once after cooling. The results were as follows:

TABLE NO. XIII

1 cc $\text{KMnO}_4 \rightleftharpoons$.005808 gm Fe

Test Ore	TiO_2	cc KMnO_4	% Fe	Deviation from 69.2%
.1398	.1*	12.20	50.68	- 8.52
.2881	.1*	32.35	65.22	- 4.00
.1578	.1*	19.65	72.32	+ 3.12
.2168	.1*	26.68	71.49	+ 2.27
.1183	.1*	13.92	68.3	- .90

* Approximate.

The following ore (Ilmenite) contained about equal amounts of iron and titanium. The weighed sample was fused with sodium carbonate and potassium nitrate after it had been treated with hydrofluoric and sulphuric acids to remove the silica. It was fused for thirty-five to forty-five minutes, the melt disintegrated in hot water, the residue washed with hot water and dissolved in hydrochloric acid. The regular Zimmermann-Reinhardt method was then used.

No results could be obtained by this method on account of the yellow color obtained when the stannous chloride was added. This was due to a small amount of platinum which came from the crucible during the nitrate fusion. In another determination no nitrate was used, but the result was again worthless.

The following samples were fused and dissolved as before, excepting that they were reduced with hydrogen sulphide and titrated, after boiling to expel the excess of hydrogen sulphide.

TABLE NO. XIV

1 cc $\text{KMnO}_4 \rightleftharpoons .005808 \text{ gm Fe}$

Sample	cc KMnO_4	% Fe
.1800	11.63	37.52
.1572	11.02	40.71
.1158	7.75	38.90
.1200	8.64	41.83
.1375	7.17	30.29
.0921	6.90	43.23

In all of the above determinations a brown precipitate separated as soon as the hydrogen sulphide was passed into the solution. This had to be filtered off because the end point was not distinct in the presence of this precipitate. A qualitative analysis of this brown precipitate showed it to contain platinum.

The sample which gave very low results was boiled for over an hour to expel the hydrogen sulphide, while the others were boiled for only about thirty minutes.

The following determinations were made according to the same procedure, but in order to remove the hydrogen sulphide a constant stream of carbon dioxide was passed through the boiling solution. This was continued also while the solution was cooling.

TABLE NO. XV

1 cc $\text{KMnO}_4 \rightleftharpoons$.005680 gm Fe

Sample	Time of Boiling	cc KMnO_4	% Fe
.1250	35 minutes	9.43	42.85
.1093	35 "	8.00	41.57
.1002	30 "	7.35	41.66
.1419	30 "	9.95	39.82
.1037	40 "	7.20	39.09
.1028	50 "	6.90	38.13
.1433	60 "	9.67	37.00
.1243	45 "	8.52	38.77
.1035	50 "	7.08	38.63
.1129	50 "	7.73	38.88
.1096	60 "	7.50	38.23
.1029	35 "	7.02	38.74

The following determinations were made by fusing the ore with sodium carbonate, dissolving the melt in hydrochloric acid, and precipitating the iron and titanium with ammonia after oxidation with sodium peroxide. This precipitate was thoroughly washed with hot water, dissolved in hydrochloric acid and titrated by the Zimmermann-Reinhardt method.

TABLE NO. XVI

1 cc $\text{KMnO}_4 \rightleftharpoons$.005637

Sample	cc KMnO_4	% Fe
.1962	13.54	39.19
.1049	7.34	39.43
.1225	8.45	38.75
.1316	9.22	38.49
.1895	13.25	39.41
.1352	9.47	39.48
.1040	7.40	40.10
.0943	6.60	39.44
.1172	8.18	39.34
.1205	8.43	39.43
.1041	7.35	39.80
.1061	7.45	39.58
.1129	7.90	39.52
.3960	27.67	39.40

The sample which ran over 40% was not washed thoroughly and there must have been some platinum left in the precipitate, for after reducing with stannous chloride and cooling a slight yellow color appeared. The precipitate of mercurous chloride was dirty. This dirty color disappeared at the end of the titration. All of the other determinations were normal.

A standard gravimetric method applied to this ore by Mr. W. J. Tilton showed it to contain 39.2% of iron.

The following determinations were made on an ore of very low iron and high titanium content. The method of procedure was the same as that used in the case of the preceding ore.

TABLE NO. XVII

1 cc $\text{KMnO}_4 \rightleftharpoons$.005555 gm Fe

Sample	cc KMnO_4	% Fe
.2298	2.10	5.07
.2731	2.34	4.76
.2456	2.15	4.86
.3045	2.50	4.46
.2388	2.05	4.76

TABLE NO. XVII (Concluded)

1 cc $\text{KMnO}_4 \rightleftharpoons$.005555 gm Fe

Sample	cc KMnO_4	% Fe
.2471	2.15	4.83
.2028	1.80	4.93
.1374	1.20	4.84
.1194	1.05	4.88

In these titrations the end point disappeared very rapidly; in fact it was very difficult to recognize the end point.

A standard gravimetric method applied to this ore by Mr. W. J. Tilton showed it to contain 3.6% of iron.

CONCLUSION

The above results show that titanium, chromium, tungsten and vanadium, if present in a solution in which iron is being determined by the Zimmermann-Reinhardt method, will cause the results to be high.



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